PATENT SPECIFICATION

Inventors: WILLIAM BERRY, EDWARD GEORGE SANDERSON and GRIFFITH ISLWYN EVANS

756,346

Date of filing Complete Specification May 13, 1954.

Application Date May 18, 1953. *No. 13942/53.*

Complete Specification Published Sept. 5, 1956.

Index at acceptance :—Classes 2(2), F2(A : G : K1), F3E : **70**, Q5B : **and 140**, E1H.

COMPLETE SPECIFICATION

ERRATA

SPECIFICATION NO. 756,346

Page 1, line 44, *for* "wraping" *read* "wrapping".

Page 1, line 49, and page 6, line 62, *for* "0°C." *read* "°C.".

Page 1, line 82, *for* "material" *read* "materials".

Page 3, line 3, *for* "ether" *read* "group".

Page 3, line 34, *for* "ben" *read* "been".

Page 3, line 88, *for* "(" *read* ")".

Page 6, line 2, *after* "film" *insert* "a".

Page 7, line 26, *for* "preparation" *read* "proportion".

Page 8, line 69, *for* "moistureproof" *read* "moistureproofing".

THE PATENT OFFICE,
28th February, 1957

DB 43343/1(18)/3671 150 2/57 R

so that the sheet wrapping material used for packaging fresh produce and processed meats should permit of appreciable passage of water-vapour therethrough, and further, that highly moistureproof sheet wrapping materials, known in variety in the art, do not fully satisfy this requirement.

To ensure the desirable degree of water-vapour transmission through sheet wrapping materials used for packaging fresh produce and processed meats, and yet to retain an adequate degree of moistureproofness of the wrapping so that the contents do not dry out, it has been found by experience that these are already known. One of the objects of such moistureproofing coatings is to render the sheet or film less permeable to water-vapour.

Moreover, British Specification No. 664,248 describes semi-moistureproof, heat-sealable sheet wrapping material, comprising a regenerated cellulose base film and a surface coating comprising a film-forming agent and a mono-, di-, tri- or tetra-ester of pentaerythritol and a saturated aliphatic mono-carboxylic acid having 12 to 35 carbon atoms in the molecule. The term "film-forming agent" is restricted therein, as regards the film-forming substance,

PATENT SPECIFICATION

Inventors: WILLIAM BERRY, EDWARD GEORGE SANDERSON and GRIFFITH ISLWYN EVANS

756,346

Date of filing Complete Specification May 13, 1954.

Application Date May 18, 1953. *No. 13942/53.*

Complete Specification Published Sept. 5, 1956.

Index at acceptance :—Classes **2(2)**, F2(A:G:K1), F3E; **70**, Q5B; **and 140**, E1H.

COMPLETE SPECIFICATION

Improvements in or relating to Semi-Moistureproof, Heat-Sealable Sheet Wrapping Material

We, BRITISH CELLOPHANE LIMITED, a British Company, of Bath Road, Bridgwater, Somerset, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention is for improvements in or relating to semi-moistureproof, heat-sealable sheet wrapping material.

Recent advances in the packaging industry have resulted in a demand for transparent heat-sealable sheet wrapping material for use for packaging fresh produce, such as soft fruits and vegetables, and processed meats, such as meat loaves. Fresh produce and processed meats usually have quite a high water-content. A highly moistureproof sheet wrapping material when used for packaging fresh produce and processed meats, may maintain a high relative humidity within the package, whereby the tendency to sliming, mould growth, and bacterial decomposition are accelerated. High relative humidities within such a package may lead also to fogging by droplet formation on the inside surface of the transparent sheet wrapping material, and thus to unattractive appearance of the package. From these considerations, it will be apparent that the sheet wrapping material used for packaging fresh produce and processed meats should permit of appreciable passage of water-vapour therethrough, and further, that highly moistureproof sheet wrapping materials, known in variety in the art, do not fully satisfy this requirement.

To ensure the desirable degree of water-vapour transmission through sheet wrapping materials used for packaging fresh produce and processed meats, and yet to retain an adequate degree of moistureproofness of the wrapping so that the contents do not dry out, it has been found by experience that these sheet wraping materials should have permeability values varying with temperature approximately as follows:—

TABLE

Temperature 0° C.	Permeability Value Range.
40	1500—6000
20	500—2000

Permeability value, at the temperature specified, is the number of grams of water-vapour which pass through 100 square metres of coated film per hour over a 24 hour period with a water-vapour differential of 50 to 55 mms. (preferably 53.4 ± 0.7 mms.) of mercury.

Sheet wrapping materials having at the temperatures specified permeability values comprised within the range hereinabove stated are referred to in the present specification as " semi-moistureproof ".

One of the objects of the present invention is to provide semi-moistureproof, heat-sealable sheet wrapping material, comprising a regenerated cellulose base film coated with a surface coating, which sheet wrapping material is particularly suited for use for packaging fresh produce and processed meats.

Sheets or films of regenerated cellulose coated with a moistureproofing coating comprising generally a cellulosic base, such as cellulose ether or ester, a gum or a resin, a wax or the like, and preferably a plasticiser are already known. One of the objects of such moistureproofing coatings is to render the sheet or film less permeable to water-vapour.

Moreover, British Specification No. 664,248 describes semi-moistureproof, heat-sealable sheet wrapping material, comprising a regenerated cellulose base film and a surface coating comprising a film-forming agent and a mono-, di-, tri- or tetra-ester of pentaerythritol and a saturated aliphatic mono-carboxylic acid having 12 to 35 carbon atoms in the molecule. The term " film-forming agent " is restricted therein, as regards the film-forming substance, to cellulose derivatives, synthetic resins, and chlorinated rubber, and to mixtures of such substances. The surface coating may com-

prise also a plasticiser and a resinous blending agent.

According to the present invention, semi-moistureproof, heat-sealable sheet wrapping material comprises a regenerated cellulose base film coated with a surface coating comprising a film-forming agent as hereinafter defined, and a waxy polymer obtainable by the polymerization of monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which contains from 12 to 34 carbon atoms inclusive.

In the present specification and claims, the term "film-forming agent" is restricted, as regards the film-forming substance, to organic solvent soluble cellulose derivatives, such as cellulose esters, e.g. cellulose nitrate (10.0% to 12.2% N_2), cellulose acetate (52% to 60% combined acetic acid), and ethyl cellulose (43% to 50% ethoxy content), and to rubber hydrochloride.

The surface coating may comprise also a plasticiser and in some cases also a resinous blending agent.

The waxy polymer ingredient of the surface coating has properties which include the following: melting point over 37° C.; insoluble (or only slightly soluble) in and lighter than water; resistant to oxidation at atmospheric temperature; possessing the characteristic physical properties of waxes including, for example, a wax-like appearance, feel and fracture and freedom from "greasiness" and "stickiness" at ordinary atmospheric temperatures; light in colour and substantially free from bituminous and resinous components.

The preferred waxy polymer ingredient is the polymer obtainable by polymerisation of monomeric *n*-octadecyl vinyl ether. This monomer has the formula:—

$$C_{17}H_{34}CH_2OCH=CH_2$$

The polymer may be prepared by reacting *n*-octadecyl alcohol with acetylene, distilling off the monomeric ether formed, and polymerising the monomeric ether with the aid of boron trifluoride. This waxy polymer has a melting point of 50° C. and a molecular weight of about 4000. The waxy material known in the trade as Gersthofen Wax V, which is essentially poly-(*n*-octadecyl vinyl ether), may be employed as the waxy polymer ingredient.

The proportion of the waxy polymer ingredient in the surface coating should lie within the range between 0.5% and 10.0%, based on the total weight of the solids in the surface coating.

The film-forming agent as hereinbefore defined may comprise 35% to 99.5% by weight of the total solids of the surface coat-

[illegible] ingredient of the surface coating with the film-forming agent. Suitable gums and resins for the purpose include, for example, gum dammar, rosin, rosin-modified glyceryl citrate alkyd resin, and ester gum. The proportion of gum or resin to be employed depends upon the gum or resin chosen, particularly with respect to its ability to blend with the waxy polymer ingredient, and upon the nature and proportion of the film-forming agent used with it. Generally speaking, however, the gum or resin may comprise 3% to 20% by weight of the total solids of the surface coating.

As the plasticiser, when presnt, there should be used a compound or mixture of compounds, having a high boiling point, which are known as "plasticisers" or "softeners" for the particular film-forming agent employed. It is preferred to use a plasticiser which is substantially odourless. Satisfactory results have been obtained with tricresyl phosphate, dicyclohexyl phthalate, and dibutyl phthalate. While the plasticiser may appear as a liquid, nevertheless it is substantially non-volatile and remains in the coating, and therefore in the present specification all the above ingredients of the surface coating are referred to, for convenience, as solids. Generally speaking, the plasticiser may comprise up to 50% by weight of the total solids of the surface coating.

The approximate limits of the proportions of the various ingredients of the solids of the surface coating may be summarised as follows, the percentages being by weight:—

Film-forming agent	-	35% to 99.5%
Waxy polymer	- -	0.5% to 10%
Gum or resin	- -	0% to 20%
Plasticiser	- - -	0% to 55%

the proportions being selected to total 100%. By varying the nature and proportions of the ingredients, it is possible to obtain a composition with wide variations in the various qualities.

The invention includes the method of producing semi-moistureproof, heat-sealable sheet-wrapping material, which method comprises applying to a regenerated cellulose base film a surface coating comprising a film-forming agent as hereinbefore defined, and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which contains from 12 to 34 carbon atoms inclusive.

In the preparation of a semi-moistureproof heat-sealable sheet wrapping material, a solid semi-moistureproof surface coating, comprising a film-forming agent as hereinbefore defined and a waxy polymer, obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long chain *n*-alkyl group of which contains from 12 to

[illegible] liquid coating composition, comprising a film-forming agent as hereinbefore defined and a

waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl ether of which contains from 12 to 34 carbon atoms inclusive, dissolved in a volatile organic solvent or solvent mixture, and thereafter removing the volatile organic solvent or solvent mixture from the applied composition by evaporation by heat at an elevated temperature, at least as high as the melting point of the waxy polymer.

The application of the liquid coating composition may be accomplished in any suitable known manner, for example by passing the base film through a bath of the liquid coating composition or by spraying the liquid coating composition on to the base film. Various coating procedures are well known in the art. Preferably the liquid coating composition is applied to the surface of the base film on both sides in a layer of such thinness that the solids remaining after evaporation of the solvent or solvent mixture produce a total coating thickness (the sum of the coating thicknesses on both sides) within the range corresponding to 1.5 gms/m² to 6.0 gms/m².

If the coated film is to be placed in direct contact with water, or if it is to be used in direct contact with products containing large amounts of water for long periods of time, it is desirable to anchor the surface coating securely to the base film. This can be done by using, as base, regenerated cellulose film which has ben impregnated with a water-dispersible urea-formaldehyde reaction product and an acid catalyst, and drying by heat. Alternatively the acid catalyst may be absent from the impregnated base film and may be included in the liquid surface coating composition. Among other proposals which have been suggested is that of anchoring the surface coating to the base film by means of an intermediate coating, e.g. of an albuminous substance such as hydrolysed gelatine whether hardened or not, or hardened casein, or of a synthetic phenol-formaldehyde resin or melamine/formaldehyde resin which is substantially unaffected by water or by the solvents used in the liquid coating composition. Suitable methods and means for enhancing anchorage are described also in British Specifications Nos. 545,523 and 593,947.

Specific methods of carrying the invention into effect will now be described by way of illustration with reference to the following examples.

Throughout the present specification, parts and proportions are parts and proportions by weight unless otherwise stated.

Example I.

Heat-sealable, semi-moistureproof sheet wrapping material was prepared by applying to both surfaces of a commercial regenerated cellulose film (thickness 0.0009") a liquid surface coating composition consisting of:—

	Parts
Nitrocellulose (11.5% N_2)	6.27
Dibutyl phthalate	2.43
Dicyclohexyl phthalate	2.60
Ester gum	1.28
Waxy polymer of *n*-octadecyl ether of vinyl alcohol (m.p. 50° C., mol. wt. about 4000)	0.22
Ethyl acetate	52.30
Toluene	34.90
	100.00

The coating operation was carried out by passing the film through a bath of the above surface coating composition at a temperature of 40° C., removing the excess of the composition from the surfaces of the film by means of doctor knives, and evaporating the solvent mixture from the applied composition by heat at a temperature of approximately 90° C. The solids remaining after evaporation of the solvent mixture produced a total coating thickness (the sum of the coating thicknesses on both sides (corresponding to 3 gms/m². As soon as the solvent had been completely removed from the applied composition, the coated film was passed through a conditioning chamber to impart moisture thereto for the purpose of restoring its original flexibility.

The finished coated film was found to be transparent and flexible, and to have a heat-seal bond strength of 232 grams when tested in air having a relative humidity of 35%, and a permeability value of 1711 at 20° C. and 4215 at 40° C. The finished coated film had good surface properties, including good slip and anti-blocking properties.

The finished coated film was admirably suitable for use for packaging fresh produce and processed meats.

Small proportions of high melting paraffin wax (m.p. 50° C. or over), or a known wax-hardener such as a high melting microcrystalline wax, may be incorporated in the liquid coating composition for the purpose of improving the slip and anti-blocking properties of the surface coating without unduly affecting the permeability value of the coated film. The high melting paraffin wax or wax-hardener, when used, may comprise up to 2.5% based on the weight of the total solids of the surface coating. This method of carrying the invention into effect is illustrated in the following example.

Example II.

Heat-sealable, semi-moistureproof sheet wrapping material was prepared in the way described in Example I, except that the liquid moistureproof surface coating was replaced by a liquid surface coating composition consisting of:—

	Parts
Nitrocellulose (11.5% N_2)	6.27
Dibutyl phthalate	2.43
Dicyclohexyl phthalate	2.60
Ester gum	1.28
Paraffin wax (m.p. 64° C.)	0.06
Waxy polymer of *n*-octadecyl ether of vinyl alcohol (m.p. 50° C., mol. wt. about 4000)	0.22
Ethyl acetate	52.27
Toluene	34.87
	100.00

The coated film was transparent and flexible, and had a heat-seal bond strength of 262 grams when tested in air having a relative humidity of 35%, and a permeability value of 1115 at 20° C. and 3112 at 40° C. The finished coated film had good surface properties, including good slip and anti-blocking properties.

The coated film was admirably suitable for use for packaging fresh produce and processed meats.

EXAMPLE III.

Heat-sealable, semi-moistureproof sheet wrapping material was prepared in the way described in Example I, except that the liquid moistureproofing surface coating was replaced by a liquid surface coating composition consisting of:—

	Parts
Nitrocellulose (11.4% N_2)	6.18
Dibutyl phthalate	2.58
Tricyclohexyl citrate	2.58
Ester gum	1.27
Paraffin wax (m.p. 64° C.)	0.08
Waxy polymer of *n*-cetyl ether of vinyl alcohol (m.p. 49° to 52° C. mol. wt. about 4300)	0.21
Ethyl acetate	52.26
Toluene	34.84
	100.00

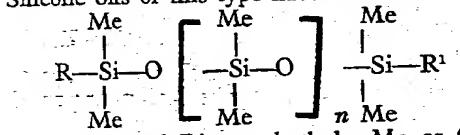
The finished coated film was transparent and flexible, and had a heat-seal bond strength of 300 grams when tested in air having a relative humidity of 35%, and a permeability value of 1000 at 20° C. and 3000 at 40° C.

The finished coated film was suitable for use for packaging fresh produce and processed meats.

In order to obtain heat-sealable, semi-moistureproof sheet wrapping material having a permeability value approaching the higher limit of 6000 at 40° C., it may be desirable, in some cases, to incorporate in the liquid surface coating composition, comprising a film-

of vinyl alcohol, the long-chain waxy group of which contains from 12 to 34 carbon atoms,

dissolved in a volatile organic solvent or solvent mixture, also (in small proportions) silicone oils of the type in which long chains of siloxane units are terminated either by organic radicals or by hydroxyl groups. Silicone oils of this type have the formula:—



wherein R and R^1 may both be Me or OH, or wherein R may be Me and R^1 may be OH. Silicone oils which have proved satisfactory for the present purpose have average molecular weights ranging from 1500 to 26000. As regards some of these silicone oils suitable for the present purpose, it would appear that cross-linking may occur between neighbouring chains. The silicone oil, when used, may be employed in a proportion of up to 2% by weight of the total solids of the surface coating. These silicone oils are soluble in the organic solvents employed.

In the claims, any silicone oil of the type described in the preceding paragraph is referred to as "silicone oil as hereinabove described".

A specific method of carrying the invention into effect, in which a silicone oil as hereinabove described is incorporated in the liquid surface coating composition, will now be described by way of illustration with reference to Example IV.

EXAMPLE IV.

Heat-sealable, semi-moistureproof sheet wrapping material was prepared by applying to both surfaces of a commercial regenerated cellulose film (thickness 0.0009") a liquid surface coating composition consisting of:—

	Parts
Nitrocellulose (12.2% N_2)	6.18
Dibutyl phthalate	2.58
Tricyclohexyl citrate	2.58
Ester gum	1.27
Paraffin wax (m.p. 64° C.)	0.15
Silicone oil (mol. wt. 26000)	0.01
Waxy polymer of *n*-cetyl ether of vinyl alcohol (m.p. 49° to 52° C. mol. wt. about 4300)	0.14
Ethyl acetate	52.25
Toluene	34.84
	100.00

The silicone oil used in the above example was that supplied by Imperial Chemical Industries Limited under the designation Silicone Oil F. 120.

The coating operation was carried out by

from the surfaces of the [illegible] means of doctor knives, and evaporating the solvent

mixture from the applied composition by heat at a temperature of approximately 90° C. The solids remaining after evaporation of the solvent mixture produced a total coating thickness (the sum of the coating thicknesses on both sides) corresponding to 3.5 gms/m². As soon as the solvent had been completely removed from the applied composition, the coated film was passed through a conditioning chamber to impart moisture thereto for the purpose of restoring its original flexibility.

The finished coated film was transparent and flexible, and had a heat-seal bond strength of 100 grams when tested in air having a relative humidity of 35% and a permeability value of 1070 at 20° C. and 4600 at 40° C. The finished coated film had good slip and anti-blocking properties.

The finished coated film was suitable for use for packaging fresh produce and processed meats.

The permeability value was determined by the method of Charch and Scroggie for measuring permeability as described in Paper Trade Journal (TAPPI Section) October 3, 1935, pages 201 to 209.

The following test has been used to measure the strength of the heat-seal bond:—

Two strips of the coated material 1½" wide are superimposed one on the other so that opposite faces of the film are in contact. A seal is made across the width of the material at one end by placing the film on a metal plate, heated to 130° C., and rolling thereover a roller ¾" wide weighted to 700 grams. The time of contact of the roller with the film is 3 seconds. The two strips so sealed are opened at the free ends. One end is gripped in a suitable clamp, and to the lower free end is attached a light balance pan by means of a suitable clamp. Weights are placed on the balance pan until the seal peels and parts. The total weight in grams required to part the seal is referred to herein as the heat-seal bond strength. A heat-seal bond strength above 60 grams is commonly accepted as satisfactory.

What we claim is:—

1. Semi-moistureproof, heat-sealable sheet wrapping material, comprising a regenerated cellulose base film coated with a surface coating comprising a film-forming agent as hereinbefore defined, and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which contains from 12 to 34 carbon atoms inclusive.

2. Semi-moistureproof, heat-sealable sheet wrapping material as claimed in Claim 1, wherein the surface coating comprises also a resinous blending agent.

3. Semi-moistureproof, heat-sealable sheet wrapping material as claimed in Claim 1 or Claim 2, wherein the surface coating comprises also a plasticiser.

4. Semi-moistureproof, heat-sealable sheet wrapping material as claimed in any of the preceding claims, wherein the waxy polymer ingredient of the surface coating is polymerised *n*-octadecyl vinyl ether.

5. Semi-moistureproof, heat-sealable sheet wrapping material as claimed in any of the preceding claims, wherein the surface coating comprises the various ingredients of the solids in proportions included within the following limits, the percentages being by weight:—

Film-forming agent	-	35% to 99.5%
Waxy polymer	- -	0.5% to 10%
Gum or resin	- -	0% to 20%
Plasticiser	- - -	0% to 55%

the proportions being selected to total 100%.

6. Semi-moistureproof, heat-sealable sheet wrapping material as claimed in Claim 5, wherein the surface coating comprises also a high melting paraffin wax or a known wax hardener in a proportion of up to 2.5% based on the weight of the total solids of the surface coating.

7. Semi-moistureproof, heat-sealable sheet wrapping material as claimed in Claim 5 or Claim 6, wherein the surface coating comprises also a silicone oil as hereinabove described, in a proportion of up to 2%, based on the weight of the total solids of the surface coating.

8. In the preparation of a semi-moistureproof, heat-sealable sheet wrapping material, the process which comprises forming upon a regenerated cellulose base film a solid semi-moistureproof surface coating, comprising a film-forming agent as hereinbefore defined, and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which contains from 12 to 34 carbon atoms inclusive, by applying to the surface of the base film a liquid coating composition, comprising a film-forming agent as hereinbefore defined and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which contains from 12 to 34 carbon atoms inclusive, dissolved in a volatile organic solvent or solvent mixture, and thereafter removing the volatile organic solvent or solvent mixture from the applied composition by evaporation by heat at an elevated temperature, at least as high as the melting point of the waxy polymer.

9. A process as claimed in Claim 8, wherein the liquid coating composition is applied to the surface of the base film on both sides in a layer of such thinness that the solids, remaining after evaporation of the solvent or solvent mixture produce a total coating thickness (the sum of the coating thicknesses on both sides) within the range corresponding to 1.5 gms/m² to 6.0 gms/m².

10. A method for the production of semi-moistureproof, heat-sealable sheet wrapping

material, which method comprises applying to a regenerated cellulose base film surface coating comprising a film-forming agent as hereinbefore defined, and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which contains from 12 to 34 carbon atoms inclusive.

11. A method for the production of semi-moistureproof, heat-sealable sheet wrapping material, substantially as hereinbefore described with reference to any one of the Examples I to IV inclusive.

12. Semi-moistureproof, heat-sealable sheet wrapping material, whenever produced by the method claimed in Claim 10 or Claim 11.

BOULT, WADE & TENNANT,
111/112, Hatton Garden, London, E.C.1,
Chartered Patent Agents.

PROVISIONAL SPECIFICATION

Improvements in or relating to Semi-Moistureproof, Heat-Sealable Sheet Wrapping Material

We, BRITISH CELLOPHANE LIMITED, a British Company, of Bath Road, Bridgwater, Somerset, do hereby declare this invention to be described in the following statement:—

This invention is for improvements in or relating to semi-moistureproof, heat-sealable sheet wrapping material.

Recent advances in the packaging industry have resulted in a demand for transparent heat-sealable sheet wrapping material for use for packaging fresh produce, such as soft fruits and vegetables, and processed meats, such as meat loaves. Fresh produce and processed meats usually have quite a high water-content. A highly moistureproof sheet wrapping material, when used for packaging fresh produce and processed meats, may maintain a high relative humidity within the package, whereby the tendency to sliming, mould growth, and bacterial decomposition are accelerated. High relative humidities within such a package may lead also to fogging by droplet formation on the inside surface of the transparent sheet wrapping material, and thus to unattractive appearance of the package. From these considerations, it will be apparent that the sheet wrapping material used for packaging fresh produce and processed meats should permit of appreciable passage of water-vapour therethrough, and further, that highly moistureproof sheet wrapping materials, known in variety in the art, do not fully satisfy this requirement.

To ensure the desirable degree of water-vapour transmission through sheet wrapping materials used for packaging fresh produce and processed meats, and yet to retain an adequate degree of moistureproofness of the wrapping so that the contents do not dry out, it has been found by experience that these sheet wrapping materials should have permeability values varying with temperature approximately as follows:—

TABLE

Permeability Value

Permeability value, at the temperature specified, is the number of grams of water-vapour which pass through 100 square metres of coated film per hour over a 24 hour period with a water-vapour differential of 50—55 mms. (preferably 53.4 ± 0.7 mms.) of mercury.

Sheet wrapping materials having at the temperatures specified permeability values comprised within the range hereinabove stated are referred to in the present specification as "semi-moistureproof".

One of the objects of the present invention is to provide semi-moistureproof, heat-sealable sheet wrapping material, comprising a regenerated cellulose base film coated with a surface coating, which sheet wrapping material is particularly suited for use for packaging fresh produce and processed meats.

Sheets or films of regenerated cellulose coated with a moistureproofing coating comprising generally a cellulosic base, such as cellulose ether or ester, a gum or a resin, a wax or the like, and preferably a plasticiser are already known. One of the objects of such moistureproofing coatings is to render the sheet or film less permeable to water-vapour.

Moreover, Specification No. 664,248 describes semi-moistureproof, heat-sealable sheet wrapping material, comprising a regenerated cellulose base film and a surface coating comprising a film-forming agent and a mono-, di-, tri- or tetra-ester of pentaerythritol and a saturated aliphatic mono-carboxylic acid having 12 to 35 carbon atoms in the molecule. The term "film-forming agent" is restricted, as regards the film-forming substance, to cellulose derivatives, synthetic resins, and chlorinated rubber, and to mixtures of such substances. The surface coating may comprise also a plasticiser and a resinous blending agent.

According to the present invention, semi-moistureproof, heat-sealable sheet wrapping material comprises a regenerated cellulose base film coated with a surface coating comprising a film-forming agent and a waxy polymer obtainable by the polymerization of

The surface coating may comprise also a plasticiser and a resinous blending agent.

The waxy polymer ingredient of the surface coating has properties which include the following: melting point over 37° C.; insoluble (or only slightly soluble) in and lighter than water; resistant to oxidation at atmospheric temperature; possessing the characteristic physical properties of waxes including, for example, a wax-like appearance, feel and fracture and freedom from "greasiness" and "stickiness" at ordinary atmospheric temperatures; light in colour and substantially free from bituminous and resinous components.

The preferred waxy polymer ingredient is the polymer obtainable by polymerisation of monomeric *n*-octadecyl vinyl ether. This monomer has the formula:—

$$C_{17}H_{34}CH_2OCH=CH_2$$

The polymer may be prepared by reacting *n*-octadecyl alcohol with acetylene, distilling off the monomeric ether formed, and polymerising the monomeric ether with the aid of boron trifluoride. This waxy polymer has a melting point of 50° C. and a molecular weight of about 4000.

The preparation of the waxy polymer ingredient in the surface coating should lie within the range between 0.5% and 10.0%, based on the total weight of the solids in the surface coating.

The film-forming agent may be a cellulose derivative such as a cellulose ester or cellulose ether, or a natural or artificial resin such as gum mastic, or a resin prepared from one or more vinyl derivatives, including styrene, vinyl acetate and vinyl chloride. The film-forming agent may comprise 35% to 60% by weight of the total solids of the surface coating, with a preferred range of 40% to 50%, reckoned on the same basis.

The resinous blending agent, when used at all, serves to blend the waxy polymer ingredient of the surface coating with the film-forming agent. Suitable gums and resins for the purpose include, for example, gum dammar, rosin, rosin-modified glyceryl citrate alkyl resin, and ester gum. The proportion of gum or resin to be employed depends upon the gum or resin chosen, particularly with respect to its ability to blend with the waxy polymer ingredient, and upon the nature and proportion of the film-forming agent used with it. Generally speaking, however, the gum or resin may comprise 3% to 20% by weight of the total solids of the surface coating.

For the plasticiser, when present, there should be used a compound or mixture of compounds, having a high boiling point, which are known as "plasticisers" or "softeners" for the particular film-forming agent employed. It is preferred to use a plasticiser which is substantially odourless. Satisfactory results have been obtained with tricresyl phosphate, dicyclohexyl phthalate, and dibutyl phthalate. While the plasticiser may appear as a liquid, nevertheless it is substantially non-volatile and remains in the coating, and therefore in the present specification all the above ingredients of the surface coating are referred to, for convenience, as solids.

The approximate limits of the proportions of the various ingredients of the solids of the surface coating may be summarised as follows, the percentages being by weight:—

Film-forming agent -	35%	to 99.5%
Waxy polymer - -	0.5%	to 10%
Gum or resin - -	0%	to 20%
Plasticiser - - -	0%	to 55%

the proportions being selected to total 100%. By varying the nature and proportions of the ingredients, it is possible to obtain a composition with wide variations in the various qualities.

The invention includes the method of producing semi-moistureproof, heat-sealable sheet-wrapping material, which method comprises applying to a regenerated cellulose base film a surface coating comprising a film-forming agent and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which comprises from 12 to 34 carbon atoms.

The invention includes semi-moistureproof, heat-sealable sheet wrapping material whenever produced by the process in accordance with the invention.

In the preparation of semi-moistureproof, heat-sealable sheet wrapping material in accordance with the invention, a liquid surface coating composition, comprising a film-forming agent and a waxy polymer obtainable by the polymerisation of a monomeric long-chain *n*-alkyl ether of vinyl alcohol, the long-chain *n*-alkyl group of which comprises from 12 to 34 carbon atoms, dissolved in a volatile organic solvent or solvent mixture, is applied to one surface or to both surfaces of the regenerated cellulose base film and the solvent or solvent mixture is removed from the applied composition by evaporation by heat at an elevated temperature, at least as high as the melting point of the waxy polymer. Various coating procedures are well-known in the art.

If the coated film is to be placed in direct contact with water, or if it is to be used in direct contact with products containing large amounts of water for long periods of time, it is desirable to anchor the surface coating securely to the base film. This can be done by using, as base, regenerated cellulose film which has been impregnated with a water-dispersible urea/formaldehyde reaction product and an acid catalyst, and drying by heat. Among other plans which have been proposed is that of anchoring the surface coating to the base film by means of an intermediate coating, e.g. of an albuminous substance such as

hydrolysed gelatine whether hardened or not, or hardened casein, or of a synthetic phenol-formaldehyde resin or melamine/formaldehyde resin which is substantially unaffected by water or by the solvents used in the liquid coating composition. Suitable methods and means for enhancing anchorage are described also in Specifications Nos. 545,523 and 593,947.

Specific methods of carrying the invention into effect will now be described by way of illustration with reference to the following examples.

Throughout the present specification, parts and proportions are parts and proportions by weight unless otherwise stated.

EXAMPLE I.

Heat-sealable, semi-moistureproof sheet wrapping material was prepared by applying to both surfaces of a commercial regenerated cellulose film (thickness 0·0009") a liquid surface coating composition consisting of:—

	Parts
Nitrocellulose (11·5% N_2)	6.27
Dibutyl phthalate	2.43
Dicyclohexyl phthalate	2.60
Ester gum	1.28
Waxy polymer of *n*-octadecyl ether of vinyl alcohol (m.p. 50° C., mol. wt. about 4000)	0.22
Ethyl acetate	52.30
Toluene	34.90
	100.00

The coating operation was carried out by passing the film through a bath of the above surface coating composition, removing the excess of the composition from the film by means of doctor knives, and evaporating the solvent mixture from the applied composition by heat at a temperature of approximately 90° C. As soon as the solvent had been completely removed from the applied composition, the coated film was passed through a conditioning chamber to impart moisture thereto for the purpose of restoring its original flexibility.

The coated film was found to be transparent and flexible, and to have a heat-seal bond strength of 232 grams when tested in air having a relative humidity of 35%, and a permeability value of 1711 at 20° C. and 4215 at 40° C.

The coated film was admirably suitable for use for packaging fresh produce and processed meats.

Small proportions of high melting paraffin wax (m.p. 60° C. or over), or a wax-hardener such as a high melting microcrystalline wax, may be incorporated in the liquid coating composition for the purpose of improving the slip and anti-blocking properties of the surface coating without unduly affecting the permeability value of the coated film. This is illustrated in the following example.

EXAMPLE II.

Heat-sealable, semi-moistureproof sheet wrapping material was prepared in the way described in Example I, except that the liquid moistureproof surface coating was replaced by a liquid surface coating composition consisting of:—

	Parts
Nitrocellulose (11·5% N_2)	6.27
Dibutyl phthalate	2.43
Dicyclohexyl phthalate	2.60
Ester gum	1.28
Paraffin wax (m.p. 64° C.)	0.06
Waxy polymer of *n*-octadecyl ether of vinyl alcohol (m.p. 50° C.; mol. wt. about 4000)	0.22
Ethyl acetate	52.27
Toluene	34.87
	100.00

The coated film was transparent and flexible, and had a heat-seal bond strength of 262 grams when tested in air having a relative humidity of 35%, and a permeability value of 1115 at 20° C. and 3112 at 40° C.

The coated film was admirably suitable for use for packaging fresh produce and processed meats.

The permeability value was determined by the method of Charch and Scroggie for measuring permeability as described in Paper Trade Journal (TAPPI Section) October 3, 1935, pages 201 to 209.

The following test has been used to measure the strength of the heat-seal bond:—

Two strips of the coated material 1½" wide are superimposed one on the other so that opposite faces of the film are in contact. A seal is made across the width of the material at one end by replacing the film on a metal plate, heated to 130° C., and rolling thereover a roller ¾" wide weighted to 700 grams. The time of contact of the roller with the film is 3 seconds. The two strips so sealed are opened at the free ends. One end is gripped in a suitable clamp, and to the lower free end is atached a light balance pan by means of a suitable clamp. Weights are placed on the balance pan until the seal peels and parts. The total weight in grams required to part the seal is referred to herein as the heat-seal bond strength. A heat-seal bond strength above 60 grams is commonly accepted as satisfactory.

BOULT, WADE & TENNANT,
111/112 Hatton Garden, London, E.C.1

Leamington Spa: Printed for His Majesty's Stationery Office.
Published at the Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.